

# Studies on preparations and analysis of essential oil from Chinese fir

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**Abstract:** Three steam distillation devices (D-1, D-2 and D-4) or one simultaneous distillation (D-3, water-diethyl ether) as well as the process of CO<sub>2</sub>-SFE (Supercritical fluid extraction) were adopted in extraction of essential oils from Chinese-fir (*Cunninghamia lanceolata* (Lamb) Hook.) and the chemical components of the extracted essential oil were analyzed by Gas chromatograph-MS analyses. The results showed that the essential oil could be almost extracted out within 2 hours and the device-3 had the highest extraction efficiency. The major chemical component of the oil was cedrol. The yield of the extracted essential oils from Chinese fir decreased gradually with the increase of the distillation time. The best condition for extraction by means of CO<sub>2</sub>-SFE is 100 kg·cm<sup>-2</sup> in pressure and 40°C in temperature for .

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## Introduction

Essential oil is a volatile, aromatic materials obtained by the steam or hydrodistillation of botanicals. Most essential oils are primarily composed of terpenes and their oxygenated derivatives. Different parts of the plants can be used to obtain essential oils, such as the flowers, leaves, seeds, roots, stems, bark, wood, etc.. The plants with the essential oils have natural resistance to fungus and termite (Chang *et al.* 1998; Huang *et al.* 2001; Lu *et al.* 1987; Shang *et al.* 2000; Shieh *et al.* 1992a). The chemical constituents of the essential oil from Chinese fir wood had been investigated (Lu *et al.* 1986, 1999; Shieh *et al.* 1992b). In this paper several preparation methods of essential oil from Chinese fir wood were carried out and the chemical components of the essential oils were analyzed.

## Materials and methods

Wood sample of Chinese fir (*Cunninghamia lanceolata* (Lamb) Hook.) came from the plantation in Dagangshan, Jiangxi Province. The sample was cut into small pieces, and then milled into powder (from 40 to 60 mesh).

GC (Gas chromatograph) -MS analyses of the essential oils were conducted by use of a HP-6890 GC system, which was equipped with a mass selective detector (MSD) as a detector and data processor. Separations were accomplished on a HP-INNOWOX 30 m×0.25 mm, 0.25µm in film thickness, and packed in a fused silica capillary column.

The temperatures of injector and detector were controlled at 250°C, helium gas was provided with the rate of 1 ml·min<sup>-1</sup>, and the temperature of oven programmed from 60°C to 250°C with a rising rate of 6°C·min<sup>-1</sup>.

Essential oils were extracted from the powder (40-200 g) of Chinese-fir wood for every 8 h by three steam distillation devices (D-1, D-2 and D-4) or one simultaneous distillation (D-3, water-diethyl ether)(Carolyn *et al.* 1977). The extraction of essential oil with device-4 was conducted hourly by using salting out-extraction for a total of seven times. In order to know the repeatability of the tests, each experiment used two parallel samples (samples 1 and 2). Meantime, the process of CO<sub>2</sub>-SFE (Supercritical fluid extraction) (Bonnie *et al.* 1988) was also adopted for extraction of the essential oils from Chinese fire within 20 min and the chemical components of the oils were analyzed by GC-MS.

## Results and discussion

### Preparation and analysis of the essential oil

Total ion chromatogram (TIC) of essential oil for Chinese fir was shown in Fig.1 and the chemical compositions of the essential oil were shown in Table 1. Yields of essential oil of Chinese-fir by four extracted methods and the contents of cedrol in the essential oils were shown in Table 2.

**Table 1. Chemical compositions of essential oil for Chinese fir**

Retention time /min	Compounds	Content /%
11.57	Thujopsene	0.99
14.53	Benzene,1-(1,5-dimethyl)-4-hexenyl	0.97
19.14	1,6,10-Dodecatrien-3-ol, 3,7,11-tr	0.97
19.99	α-Thujaplicin	1.03
20.28	Cedrol	55.18
20.98	1H-3a,7-Methanoazulene,2,3,4,7,8	0.25
21.80	α-Bisabolol	1.17
21.99	Hinokitiol	0.89
27.93	1-Naphthalenepropanol, alpha.-eth	1.78

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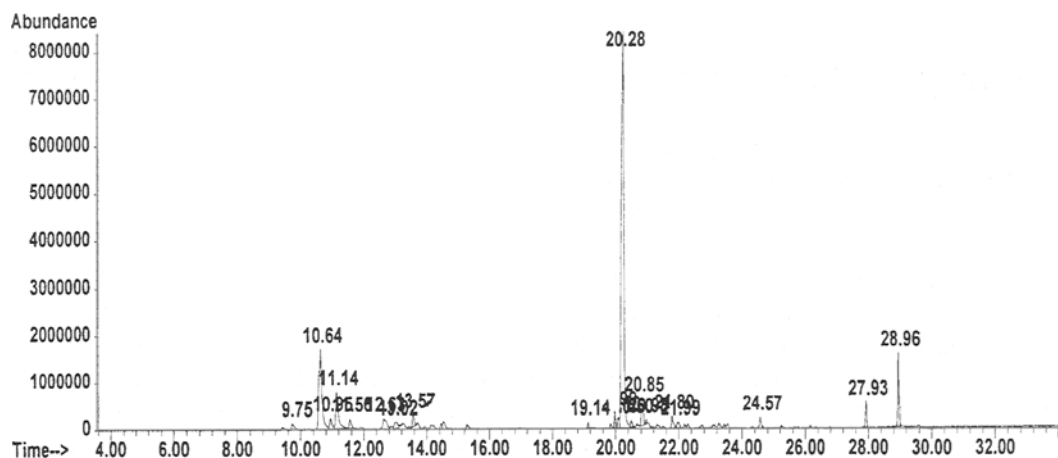


Fig.1 Total ion chromatogram of essential oil of Chinese fir

Table 2. Yields of the essential oils and contents of cedrol in the oils

Devices	Sample	Yield <sup>a</sup>	Content of cedrol <sup>b</sup>
1	1	1.05	43.11
	2	2.61	58.24
2	1	2.40	68.85
	2	1.92	62.10
3	1	2.97	64.98
	2	3.11	74.43
4	1	2.98	55.18

Note: a: (Ext. oil/oven dried wood)X100 (w/w); b: percent in peak area of TIC.

From Table.2, we can see that the yield of essential oil of Chinese-fir extracted by using the device-3 (2.97% and 3.11%) was highest than those of other three devices. GC-MS analysis of the oils showed that major chemical component was cedrol (Lu *et al.* 1986) and its content was in range of 43.11%-74.43%.

#### Extraction of essential oils with device-4 in different time by using salting out-extraction

The yields of essential oils extracted with device-4 and salting out-extraction in different distillation time (one times per hour for total of seven times), were shown in Table 3. The yield gradually decreased as the increase of the distillation time. And the total yields of samples 1 and 2 (5.62%) were higher than that (2.98%) of the extraction method without salt. The essential oil was almost extracted out within 2 hours under this condition. The contents of cedrol in the essential oils were above 49% for the first 3 times of extraction and below 42% for later 4 times of extraction under this condition.

Table 3. Yields of the oils extracted with Dvice-4 and content of cedrol in the oils at different distillation times

Distillation time /h	Samples	Yield <sup>a</sup>	Content of cedrol <sup>b</sup>
1	1	1.96	57.41
	2	2.01	49.54
2	1	1.48	86.14
	2	1.09	87.07
3	1	0.43	57.88
	2	0.58	66.11
4	1	0.50	25.50
	2	0.26	41.88
5	1	0.44	31.09
	2	0.22	34.42
6	1	0.26	19.57
	2	0.18	25.08
7	1	0.34	22.27
	2	0.24	12.09
Total mean yield		5.62	

Note: a: (Ext. oil/oven dried wood)X100; b: percent in area of TIC.

#### Extraction of essential oils by means of supercritical fluid extraction (SFE)

The process of supercritical fluid extraction (SFE) had an important position on the chemical and food industry in recent years. It has some distinct advantages over other separation techniques: 1) Thermally unstable compounds can be separated at low temperature; 2) the solvent can be removed easily from the solute by reducing the pressure and/or adjusting the temperature; 3) Thermal energy requirements are lower than that for distillation; 4) Surprisingly high selectivity for the solute can be accomplished; 5) Rapid extraction can be achieved due to low viscosity, high diffusivity and solvating power of the supercritical fluid solvent. Carbon dioxide as a supercritical fluid solvent is attractive since it is nontoxic, nonflammable, inert, readily available in high purity, inexpensive, and then has low surface tension and viscosity, and high diffusivity (Bonnie *et al.* 1988).

Essential oils were extracted from Chinese-fir wood by

CO<sub>2</sub>-SFE process within 20 min and were analyzed by GC-MS. The results were shown in Table 4.

From Table 4, it was shown the pressure of 100 Kg·cm<sup>-2</sup> and temperature 40°C was the best condition (sample 3) for the oil extraction of the Chinese-fir wood by CO<sub>2</sub>-SFE process.

**Table 4. The results of test of SFE (supercritical fluid extraction)**

Samples	Pressure /Kg·cm <sup>-2</sup>	Temperature /°C	Yield <sup>a</sup>	Content of cedrol <sup>b</sup>
1	300	40	1.83	56.18
2	200	40	2.56	40.71
3	100	40	3.14	48.23
4	80	40	1.26	50.91
5	65	30	0.34	35.47

Note: a: (Ext. oil/oven dried wood)X100; b: percent in area of TIC.

## Conclusion

The yield of essential oil of Chinese-fir extracted by using the device 3 was highest than those by using other three devices. GC-MS analysis of the oils showed that major chemical component was cedrol in range of 43.11%-74.43%.

The yield of essential oil gradually decreased as the increase of the distillation time. Also the results revealed that the essential oil was almost extracted out within 2 hours under this condition.

The best conditions for extraction of essential oil from Chinese-fir wood by means of CO<sub>2</sub>-SFE were 100 Kg·cm<sup>-2</sup> in pressure and 40°C in temperature.

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